THE DEVELOPMENT OF A HIGH VACUUM SYSTEM

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THIN FILMS RESEARCH LABORATORY
TECHNICAL REPORT NO. 1
October, 1965

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PREFACE

The Thin Films Research Laboratory wishes to express its appreciation to the Western Electric Company for the gift of the original vacuum system; to Professor F. W. Tatum of the Department of Electrical Engineering for the support of student help, for the allocation of space, and for funds for the gauge; to Dean Claude Albritton whose initial support through a "seed money" grant made the project possible; to the National Science Foundation for assistance through the undergraduate research participation program; and to the National Aeronautics and Space Administration for contribution (under Grant NGR 44-007-006) to the final phases of this work.

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INTRODUCTION

Two years and six months ago, a well-worn production line vacuum pumping cart was presented to Southern Methodist University by Western Electric.

Although there was an urgent need for a good vacuum system for the initiation of thin films research, the condition of the components on the cart and the smallness of the diffusion pump caused some doubt as to whether this system ever could be reconstructed for use in the regular evaporation of metals at 10^{-6} Torr in a vacuum chamber of reasonably large working volume.

This report describes the gradual evolution of this vacuum cart into the present vacuum system in use in the Thin Films Research Laboratory. Improvements made since this cart was received have made it possible to obtain in 20 hours an ultimate vacuum of 2.8×10^{-8} Torr. In a cylindrical working volume eleven inches in diameter and thirteen inches high, a vacuum of 8.0×10^{-8} Torr has been achieved in a 2-hour period.

Details of the original system and the process of modification are given in the following sections.

THE ORIGINAL SYSTEM

On March 21, 1963, work began on the design and fabrication of a usable laboratory pumping system from a Distillation Products, Inc.* High Vacuum Pumping Unit, Model No. SE 38-0135[1]**. This unit was made in 1953 or 1954 to evacuate magnetron tubes on a production-line basis. The unit was on casters and was pulled through a bake oven by a conveyor so that the tube would be baked and pumped at the same time[2]. Individual parts of the unit consisted of: a Distillation Products, Inc., MCF 60-08 diffusion pump with integral valving mechanisms; a Kinney CVM 3153 Compound Mechanical Vacuum Pump with motor; a General Electric Model 21SA2AM2 Freon refrigeration unit; and a Phillips-type ionization gauge with control.

The diffusion pump is a nominal two-inch pump, with a capacity of 60 liters per second. It is a three-stage fractionating type pump with provisions for water cooling. The integral valving mechanism consists of a main-gate valve, a fore-arm valve, and a roughing valve. These valves are arranged within a manifold that is affixed to the top of the pump. The valves are all of the electrically actuated solenoid type. The pump boiler is heated with a 200-watt, 220-volt AC, pancake heating element that attaches under the pump.

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*Now Consolidated Vacuum Corporation (CVC)
**The List of References is on Page 23.

The mechanical vacuum pump or fore-pump is a two-cubic-feet-per-minute, two-stage rotary piston pump. A three-phase 1/4 H.P., 220 VAC supplied the original power for this pump, but it has been replaced by a single phase, 1/3 H.P., 115 VAC motor. Initial tear-down and inspection of this pump revealed slight scoring on the piston slides and bearings. The pump was rebuilt and resealed and will maintain a vacuum of 3×10^{-3} Torr.

The purpose of the refrigeration unit is to chill a cylindrical surface located in the throat of the diffusion pump, but above the gate valve. This cold surface helps prevent back-streaming of pump fluid vapors into the vacuum chamber. The expansion valve has been adjusted to maintain a temperature of -40° C. The operating fluid is "Freon 12" and the compressor is rated at 1/2 H.P., 115 VAC. A valve is provided that allows hot, high pressure Freon to flow through the cold surface when a rapid defrosting cycle is needed.

The vacuum gauge is a standard Phillips type of cold cathode ionization gauge. This gauge is often known as a "Penning" gauge. Vacuum is measured in four ranges covering from 0.5 to 1×10^{-6} Torr. Both the gauge tube and the electronics of this unit required extensive repair, and results are still not ideal although the gauge is usable.

Additional items consisted of the cart and wheels, a control panel with switches and relays, and various valves and brackets associated with the system.

MODIFICATION OF THE SYSTEM

A decision was made to disassemble the original vacuum system and then to build up each of the four basic units as individual components. These components would then be used as building blocks in the design of a highly flexible system. Some idea of the system capabilities had to be obtained first, however, before a decision could be made as to whether this build-up would be economically feasible.

A vacuum "run" was made on May 13, 1963, after the original system was placed in operation. Repairs up to this date had consisted of a tear-down inspection and a cleaning. New fluid was placed in both the mechanical pump (Welsh Duo-Seal Oil) and the diffusion pump (CVC Convoil 20). The refrigeration system required a new charge of Freon. Electrical changes were made that allowed operation on 220 VAC single-phase current. The results of this run are shown in Figure 1. This run was performed with a ten-inch aluminum base plate, and with an eight-inch bell jar as a vacuum chamber. Gauging was performed by a borrowed CVC cold cathode ionization gauge* with recent calibration. The results of this run and of similar runs were enough to establish that the system could meet the required specifications of 4×10^{-6} Torr in two hours.

*The gauge unit was generously loaned by Mr. Dale Roberts, Field Engineer for CVC.

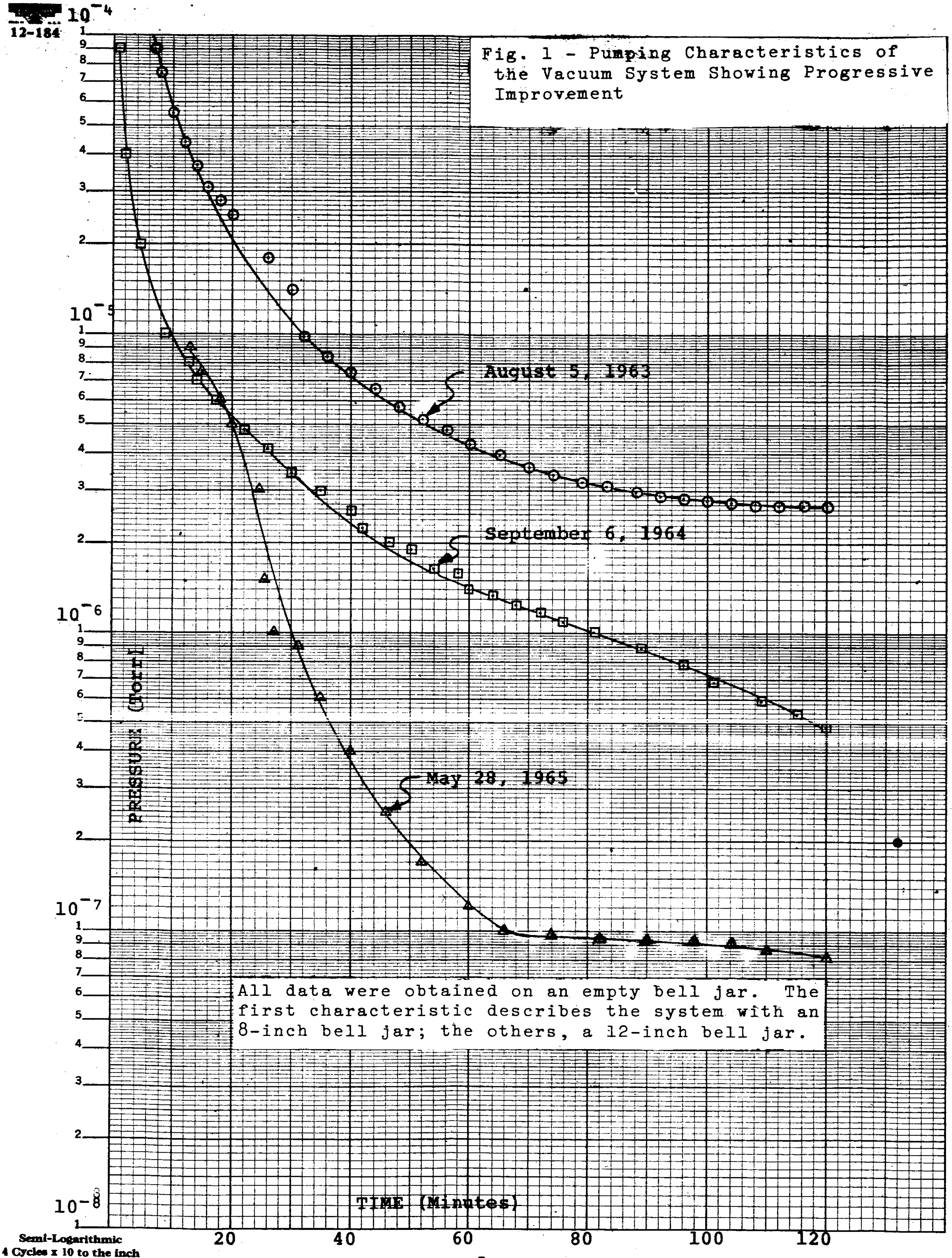


Fig. 1 - Pumping Characteristics of the Vacuum System Showing Progressive Improvement

The system was again torn down to facilitate the construction of the individual building blocks. The fore-pump was placed on a separate base with a motor and a fail-safe valve. (This valve closes the vacuum line if power is lost.)

The refrigeration system was placed on a base and was fitted with flexible lines to carry Freon to the diffusion pump throat area. The diffusion pump was fitted with a fifteen-inch-diameter stainless steel baseplate and was mounted in a suitable bench. This bench was made of slotted steel angle and Formica-covered plywood.

Formica-covered wood was used to construct a system control panel that included all pumping controls, two gauge circuits, and a current supply for resistive heating of an evaporation source.

A bell jar of twelve-inch inside diameter and fifteen-inch height and a neoprene bell jar seal were obtained. A bell jar guard was constructed of expanded steel mesh and stainless steel strips, and a counter-weighted hoist was erected to lift the bell jar. (See Figure 2.)

Baseplate feed-throughs were designed and constructed for the following purposes: high current supply, Phillips gauge, thermocouple gauge, transmission of rotary motion, voltage measurement, and air admission. Drawings of these feed-throughs are shown in Figures 3-7. All feed-throughs were designed in the laboratory for this particular system. Feed-through material was limited to stainless steel and OFHC copper, with neoprene as a sealing agent.

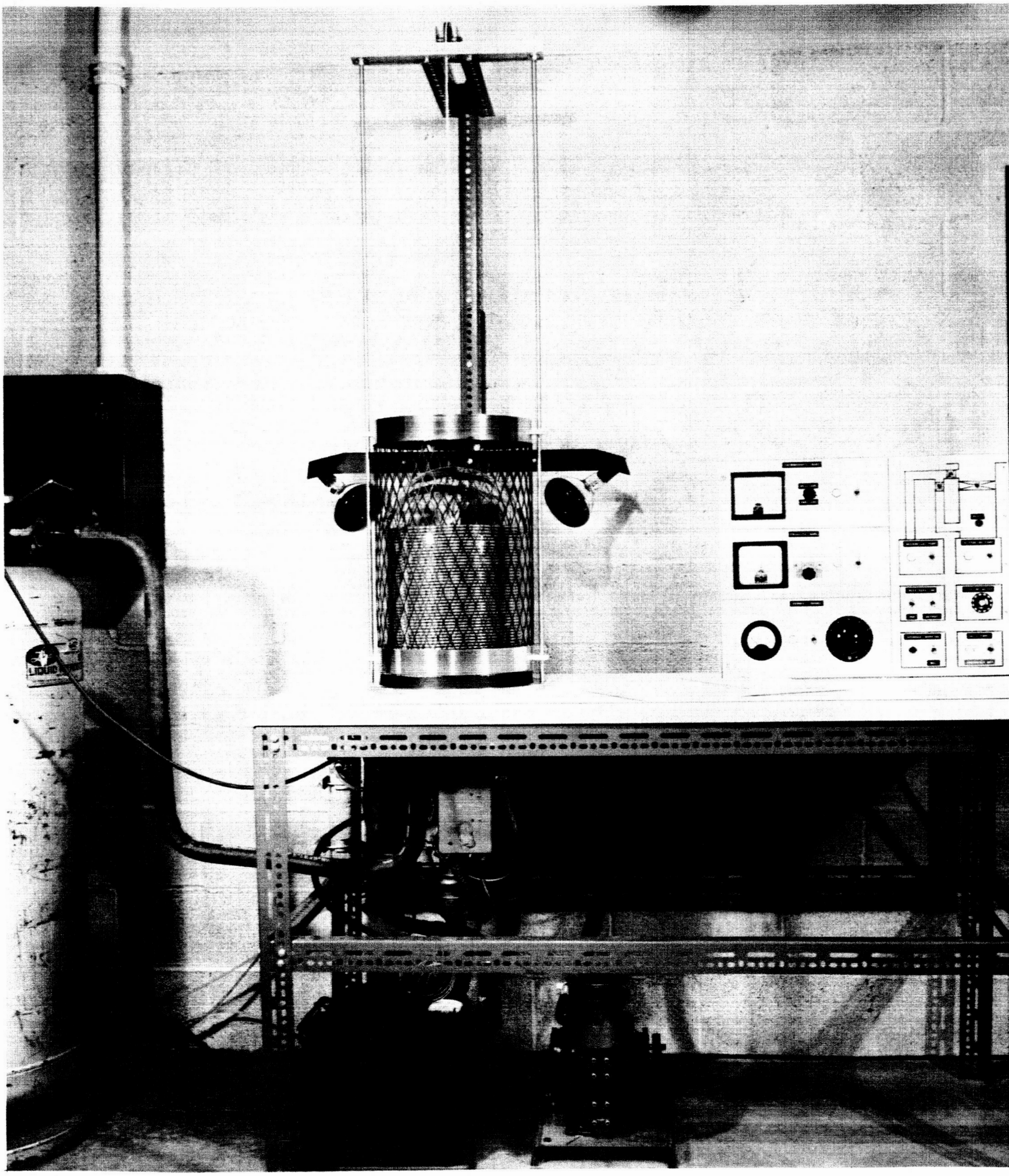


Fig. 2. Vacuum system, with control panel and hoist.

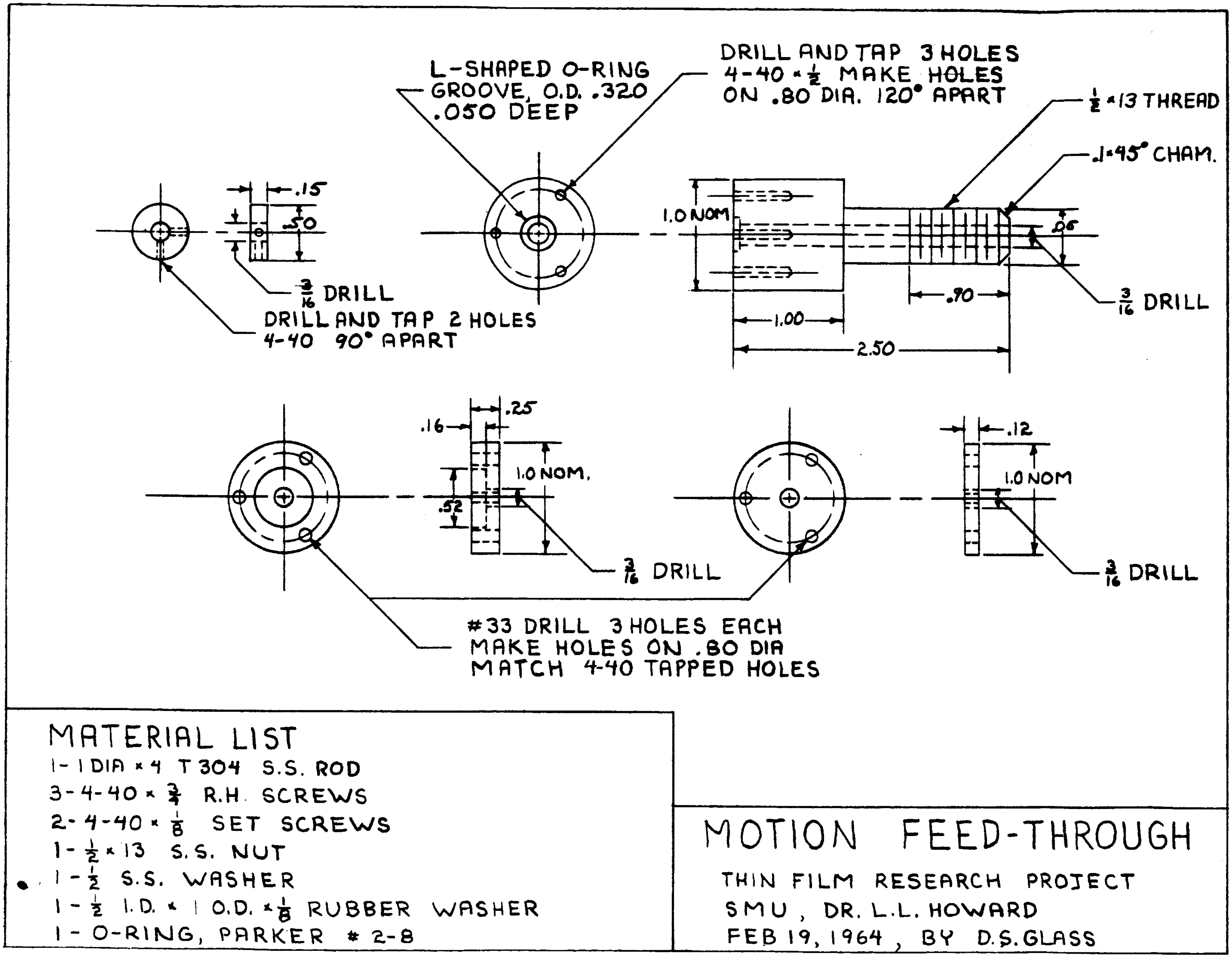


Fig. 3. Motion Feed-Through

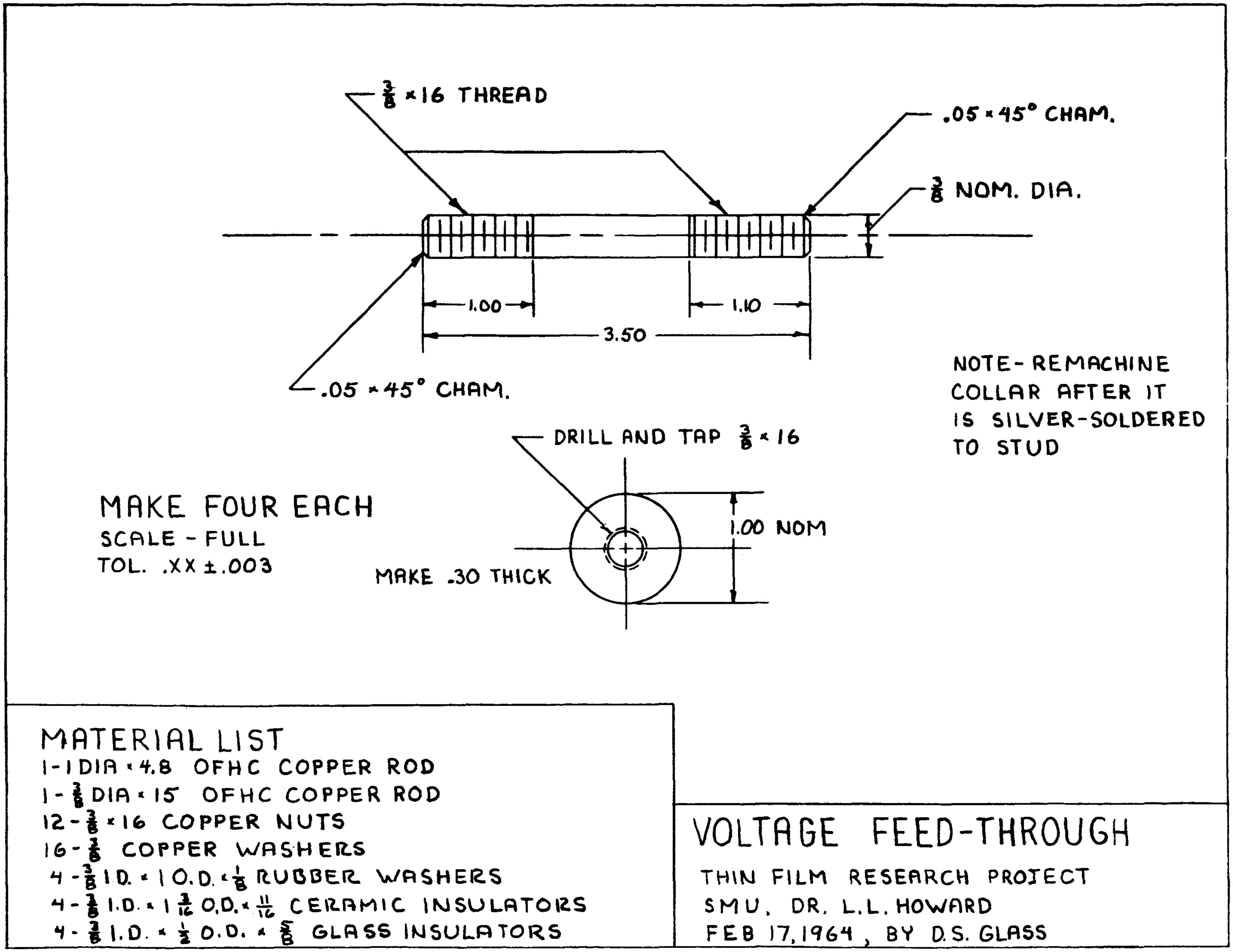


Fig. 4. Voltage Feed-Through

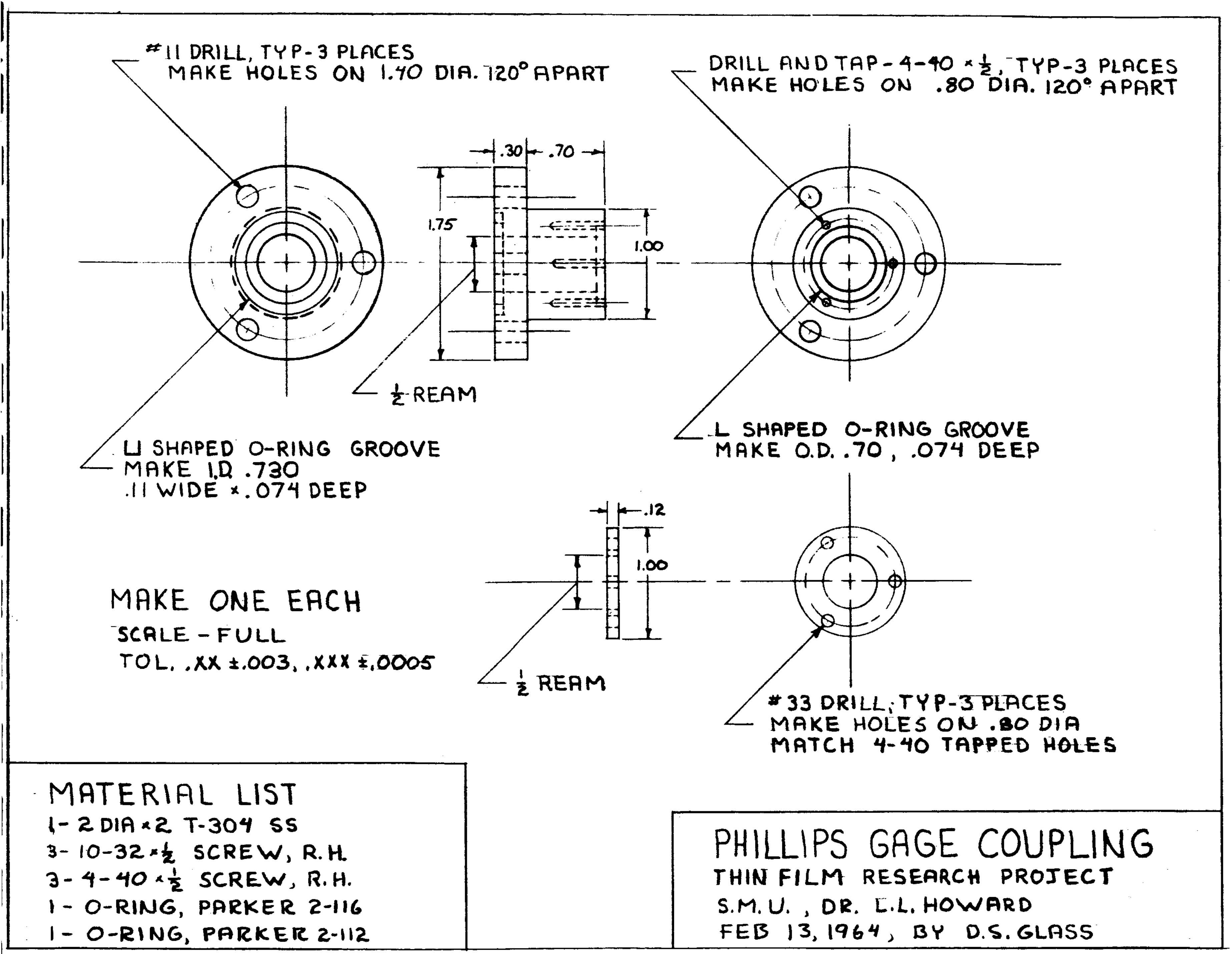


Fig. 5. Phillips Gauge Coupling

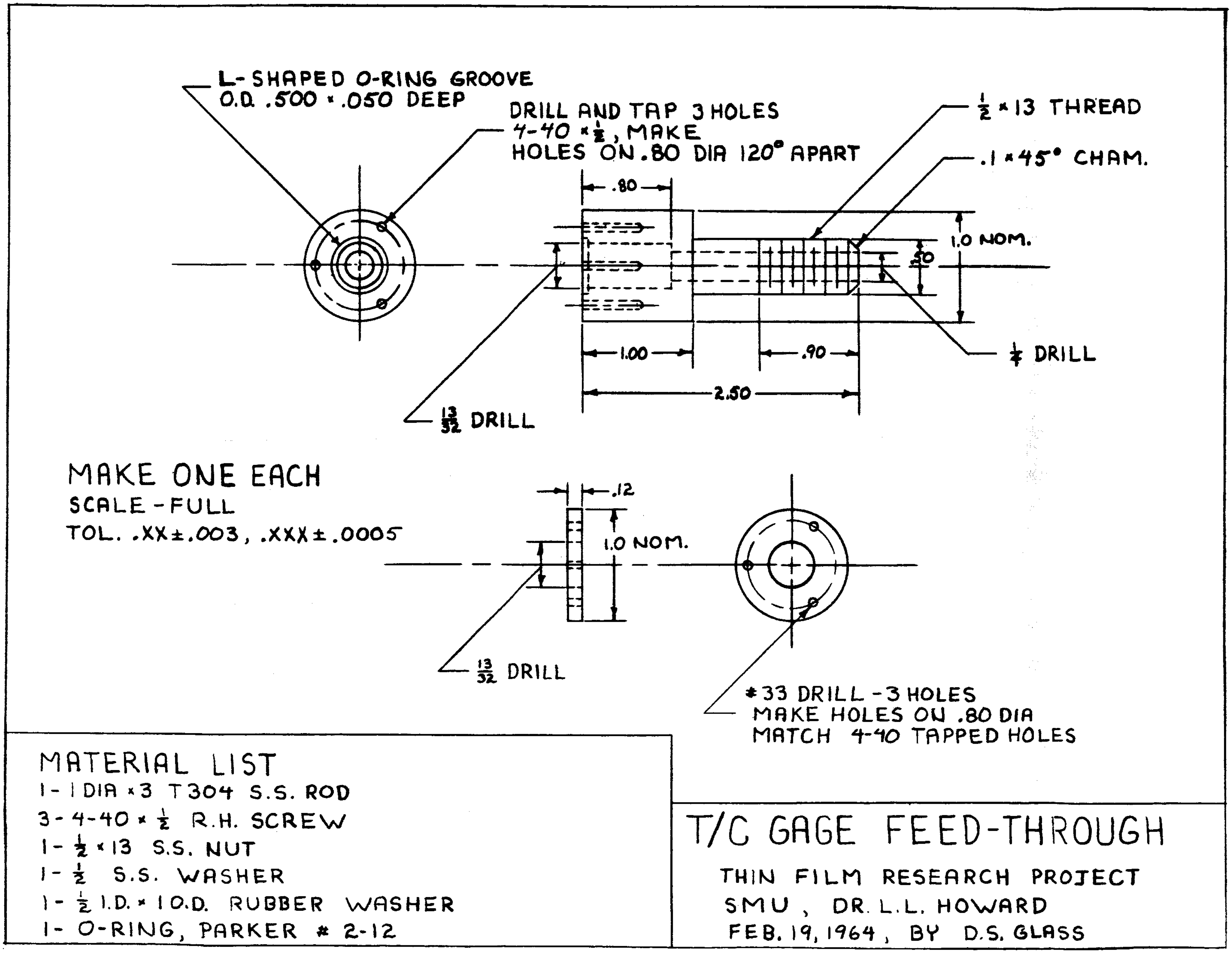
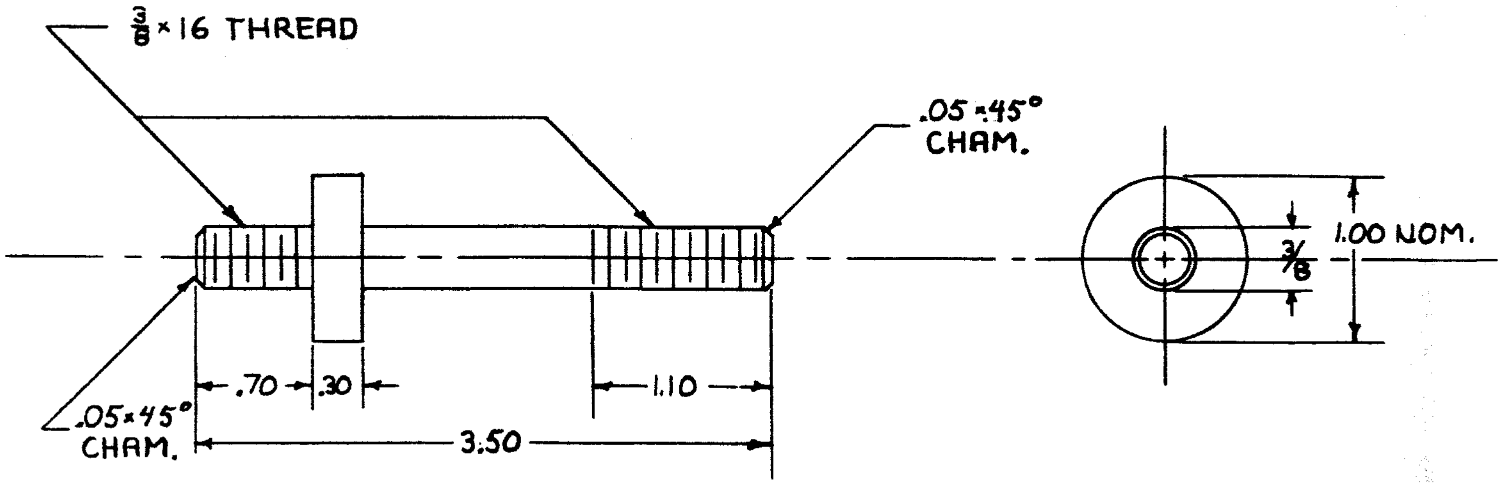


Fig. 6. Thermocouple Gauge Feed-Through



MAKE TWO
SCALE - FULL
TOL .XX ± .003

MATERIAL LIST
1- 1 DIA × 7.20 OFHC COPPER ROD
6- 3/8 × 16 COPPER NUTS
8- 3/8 COPPER WASHERS
2- 3/8 I.D. × 1 O.D. × 1/8 RUBBER WASHERS
2- 3/8 I.D. × 1 3/16 O.D. × 11/16 CERAMIC INSULATORS
2- 3/8 I.D. × 1/2 O.D. × 5/8 GLASS INSULATORS

100 AMP FEED-THROUGH
THIN FILM RESEARCH PROJECT
S.M.U., DR. L.L. HOWARD
FEB 14, 1964, BY D.S. GLASS

Fig. 7. 100-Ampere Feed-Through

The control panel and gauges are designed to control the entire pumping process. Pilot lights and power switches are provided for main line power, the diffusion pump, and the mechanical pump. Refrigeration is controlled with a power switch and a defrost switch. Diffusion pump cooling water is controlled with a needle valve. Emergency over-heat circuits consist of a switchable warning bell and an automatic diffusion pump boiler shut-down relay. This circuit is triggered if the cooling coil temperature exceeds 90° C. Valve operation is performed by toggle switches located on a pictorial representation of the system drawn on the panel. The entire control panel is hinged on the right edge and may be opened for access to all control circuits and systems.

Vacuum gauging is done with two different gauges. A thermocouple gauge circuit was constructed using two CVC gauge tubes. One tube is mounted on the base plate to monitor system pressure. The other tube is affixed to the diffusion pump fore-arm to measure vacuum produced by the mechanical pump. Thermocouple gauges are sensitive from 1 to 1×10^{-3} Torr, and are used during the rough pumping process. The original Phillips gauge was built back to specification and was mounted in the control panel. A rough calibration of this gauge was performed by comparing the readings of another gauge with those of the Phillips gauge. The Phillips gauge still is used, but only for a rough indication of vacuum.

The current source for heating metals to be evaporated consists of a ten-ampere Variac, a meter, and a heavy duty 1-to-20 current transformer. This system is capable of producing 120 amperes without over-heating.

All of the above construction, modification, and addition was complete by September of 1964. System characteristics at this time also are shown in Figure 1.

The system was used for extensive material testing while the above work was in progress. The purpose of this testing was to determine the suitability of certain materials for use in the system and to monitor the progress of the reconstruction. The design and construction of fixtures to be used for bismuth film evaporations also took place during this time.

Two new additions were made to the system in late 1964. Stainless steel bell jar guide rods were added to place the jar accurately on the baseplate. An infrared bake-out system was assembled for aiding pumping speed after the system is open to air for a considerable time.

In December of 1964, it was determined that bismuth could be evaporated in this system in the low 10^{-6} Torr range. This was established by extensive testing with the evaporation of bismuth onto glass slides while monitoring current, pressure, and film resistance. Up to this time, there was considerable doubt as to whether such a large vacuum chamber could be pumped adequately with a two-inch diffusion pump. These tests established limited capability.

A decision was made in January of 1965 to optimize the pumping system by three methods: (1) the replacement of all neoprene seals with Viton "A" O-Rings; (2) the replacement of the diffusion pump fluid with Dow-Corning DC-705 Silicone Fluid; and (3) the installation of a liquid-nitrogen-cooled shroud in the chamber. At this time also, funds were received that allowed the purchase of an effective gauging system, a Varian Hot Filament Bayard-Alpert-Type Ionization Gauge and Control. (This gauge accurately measures pressure to 1×10^{-10} Torr.)

The new seals were installed because Viton "A" exhibits virtually no outgassing at pressures in the 10^{-8} Torr region. DC-705 fluid was chosen because of its low vapor pressure in the 10^{-8} Torr region. Another advantage of DC-705 is that it will not deteriorate rapidly (as most diffusion pump fluids do) if accidentally exposed to atmospheric pressures (always a possibility in a vacuum system).

The liquid-nitrogen shroud was constructed because of the success of the "cold-trapping" method[3] in markedly reducing pressure. It was felt that the shroud should lower evaporation pressures at least one decade. The shroud took the form of an eleven-inch (inside diameter) coil of 1/4-inch OD copper tubing. Fifty coils made a cylinder 12.5 inches tall and used 150 feet of tubing. Coil support was provided with three stainless steel legs ground to a knife point at the supporting edge. Liquid-nitrogen feed-throughs were constructed of thin wall, stainless steel tube with the copper line passing down the middle. (See Figure 8.) Both the feed-throughs and the knife-edge supports were designed to minimize heat transfer from the base plate. The coil was wound on a unique wooden form (see Figure 9) in a lathe. Silver solder was used to connect adjacent coils while they were still on the form. The form was then disassembled and removed.

Note that no liquid-nitrogen connections are made inside the vacuum chamber. The liquid nitrogen feed-throughs are removed with the coil, and all connections are made outside the bell jar. (See Figure 10.) Liquid nitrogen is supplied from a 110-liter self-pressurized dewar. An insulated line carries liquid to the bottom of the coil; an exit line is vented to air. Nitrogen flow is regulated to provide a very slight trickle of liquid at the exit vent. Figure 1 also shows the system capabilities with the shroud in operation.

Two methods have been devised for shroud operation. If fast pump-down is desired, then the nitrogen is turned on when the diffusion pump is switched to "open". If lowest system pressure is desired, then the nitrogen is allowed to flow only after the lowest possible pressure obtainable by the diffusion pump has been reached.

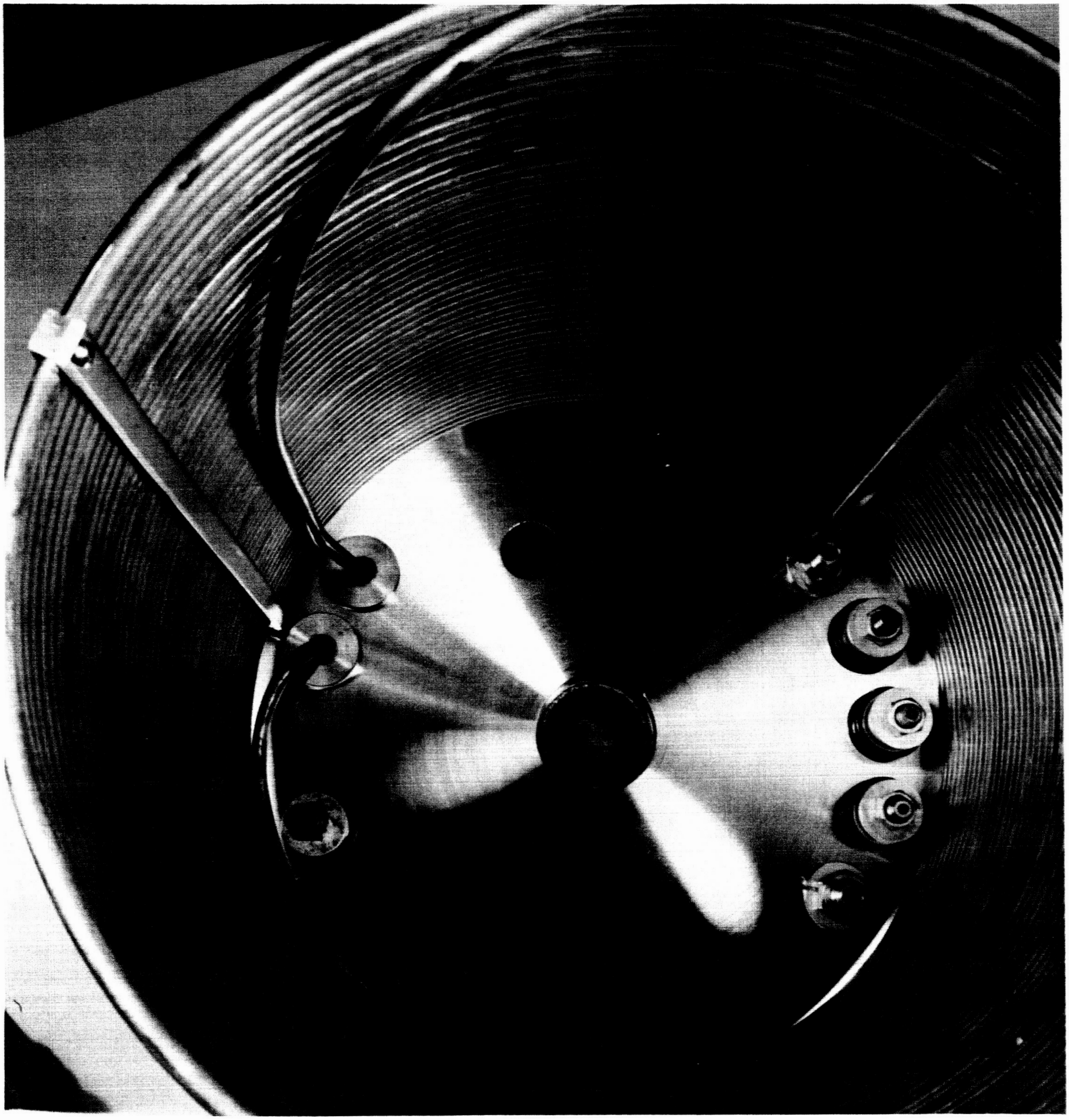


Fig. 8. View looking toward the baseplate from inside the top of the liquid nitrogen shroud.

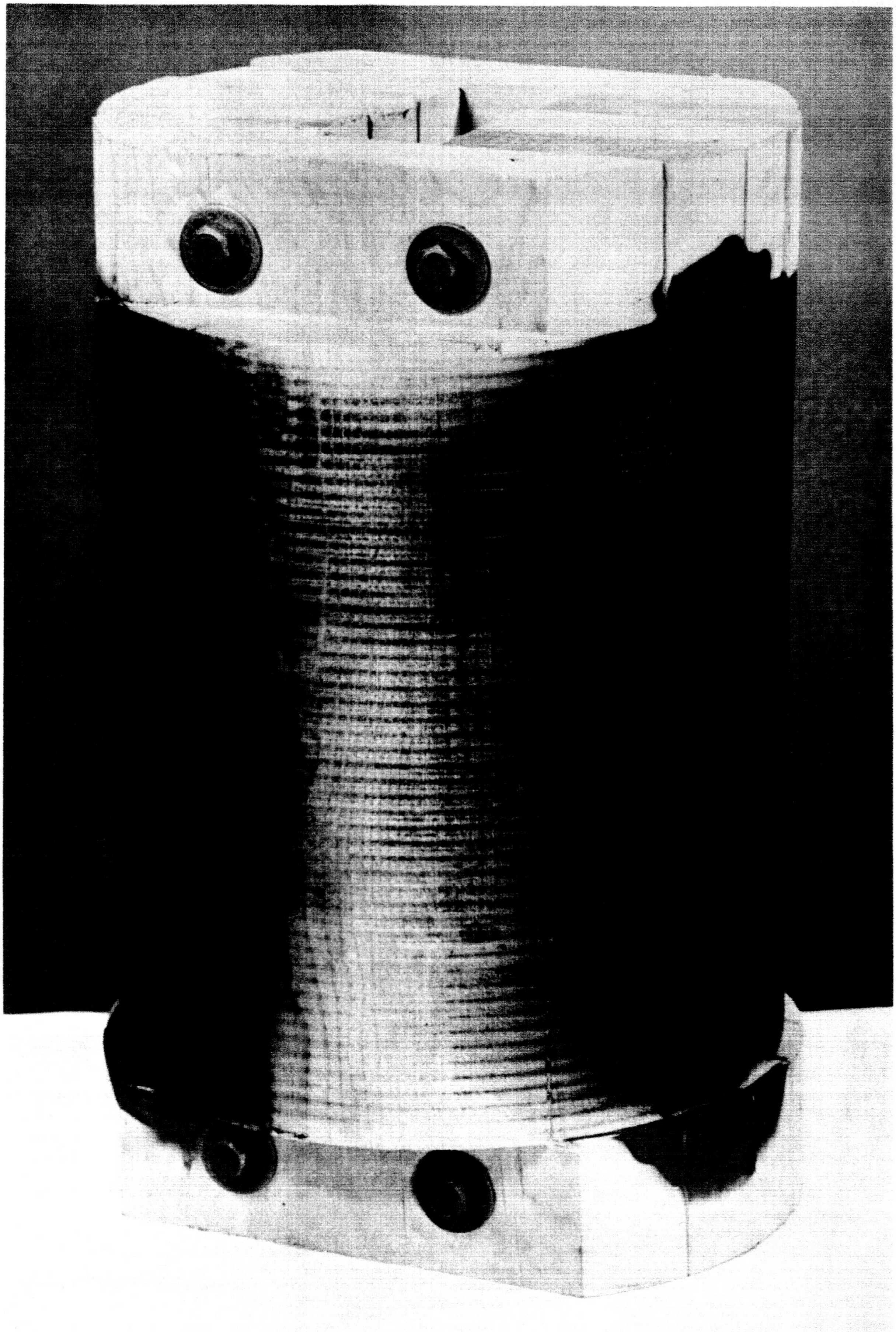


Fig. 9. Coil form for fabrication of the liquid nitrogen shroud.

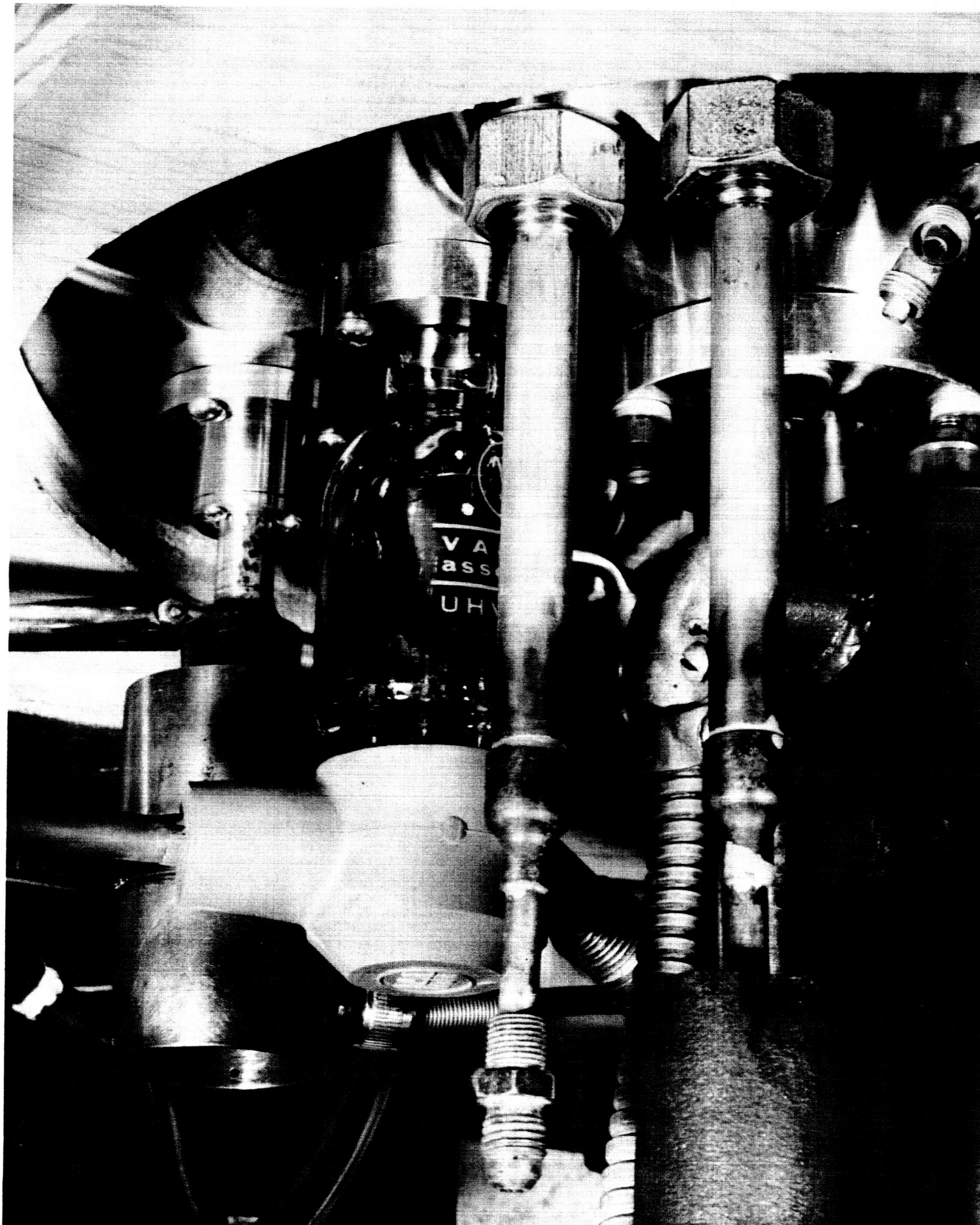


Fig. 10. View below the baseplate. The two tubes in the foreground are the liquid nitorgen feed-throughs.

An ultimate pressure (in an empty bell jar) of 2.8×10^{-8} Torr has been reached with an overnight pump-down followed by three hours of liquid-nitrogen pumping.

These last figures represent the present capability of the vacuum system in the Thin Film Laboratory. It is felt that this is the best vacuum obtainable and any further attempts to improve vacuum in this system would not be economically worthwhile. Expenditures to date are given on Page 22 and are approximately 12% below the original estimate. (The ionization gauge was not in this estimate.)

It is somewhat amazing that this system will do so well as it does since it is composed of components that are well over ten years old and have seen considerable usage. Figure 11 shows the system as it is used currently, with the gauging and film resistance control rack at the left. Evaporations beginning at 4×10^{-7} Torr are now routine.

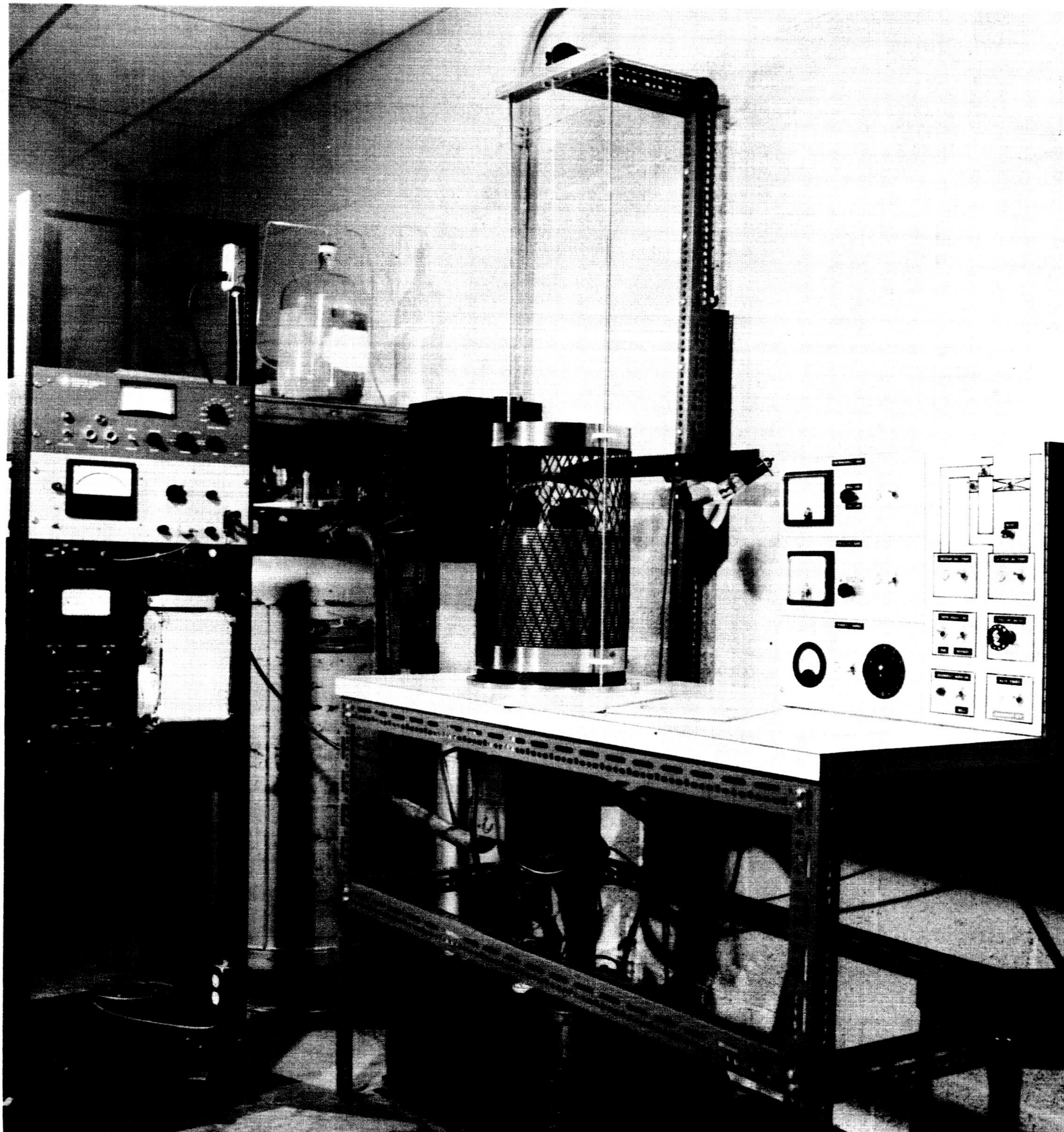


Fig. 11. Complete system. Rack to the right contains the ionization gauge control unit and film resistance monitoring system. Liquid nitrogen supply for the shroud is in the rear to the right of the rack.

MAJOR EXPENDITURES

Diffusion Pump Repair and Build Up	$ 51.00
Mechanical Pump Repair and Build Up	10.00
Refrigeration Repair and Build Up	55.00
Table Material	64.00
Stainless Steel Base Plate	125.00
Pyrex Bell Jar	60.00
Thermocouple Gauge Parts	40.00
Varian Ionization Gauge	625.00

LIST OF REFERENCES

1. Distillation Products Incorporated
   Drawings No. SE 38-0135 Layout
   SE 38-1219 Plumbing
   SE 38-1152 Electrical

   These prints are on file with Consolidated Vacuum Corporation, Rochester, New York.

2. "Applications of the 'In-Line' Exhaust Principle," by H. Glynn Warren, Vacuum Symposium Transactions, 1954.

3. "A Versatile Ultra-High Vacuum System for Thin Film Research," by C. Robert Meissner, Vacuum Symposium Transactions, 1960.